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Bis(4-chlorophenyl)(5-pyrimidinyl)methanol

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Abstract

The title compound, $C_{17}H_{12}Cl_2N_2O$, has a twisted conformation with the following angles between the three planes in the molecule: 86.0 (4)° between the two p-chlorophenyl groups [planes 1 and 2], 85.0 (4)° between plane 1 and the pyrimidine ring (plane 3), and 90.5 (4)° between planes 2 and 3. There is an intermolecular hydrogen bond [O···N 2.847 (4) Å and O—H···N 161 (5)°].

Comment

The title compound (LY43578), (1), is very similar in structure to both parinol, (2) (Kennard et al., 1981),

and fenarimol, (3) (Albinati et al., 1988), which show fungicidal activity as a result of inhibition of a P450 enzyme. Parinol, (2), has a pyridine group instead of the pyrimidine heterocycle in the title compound. Fenarimol, (3), is a structural isomer of the title compound, where one of the two Cl atoms is in an ortho position rather than the para position of the title compound. Following reports that certain nitrogen heterocyclic antifungal agents were effective inhibitors of another P450 enzyme, aromatase (Taylor et al., 1987; Mason et al., 1985), the title compound was synthesized as part of a structure–activity study in which inhibitors for the P450–aromatase system were developed (Jones et al., 1990). It shows moderate activity as a P450–aromatase inhibitor (Jones et al., 1990) and is related to LY113174 (Caruso & Rossi, 1998) and LY56110 (Lindstrom & Whitaker, 1987; Hirsch et al., 1987).

(1)   (2)

(3)
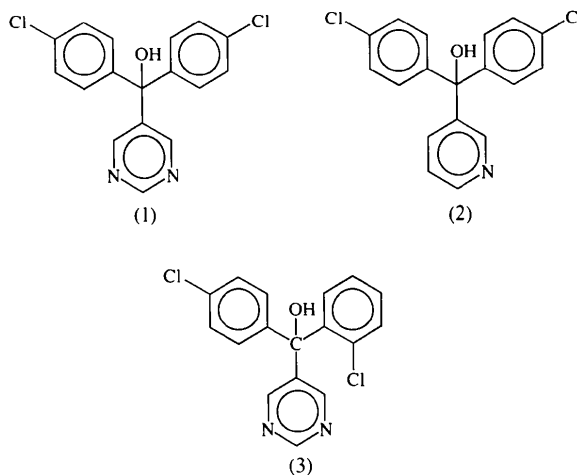
The bond distances and angles in the molecule, including the single bonds involving C(17) and the aromatic bonds, show no unusual features. The expected phenyl-ring bond-angle distortion pattern caused by Cl-atom substitution is seen (Brisse & Sygusch, 1974; Domenicano & Murray-Rust, 1979). There is no significant deviation of any atom from the three principal planes of the molecule, deviations which include Cl(1) at 0.036 (4) Å and Cl(2) at 0.059 (4) Å from their respective planes. The O—C_{tet}—C_{ipso}—C_{ortho} torsion angles, which show the relationship between the four substituents on the C(17) atom, are listed in Table 1.

The analogous O—C_{tet}—C_{ipso}—C_{ortho} torsion angles for parinol, (2), are: 155.1 (3) and −23.3 (3)° for the first p-chlorophenyl ring, 155.9 (3) and −20.4 (3)° for the second p-chlorophenyl ring, and −78.2 (3) and 91.5 (3)° for the pyridine ring. Whereas the title compound has angles between the planes of the rings close to 90°, in parinol, these angles are 77° between the two p-chlorophenyl rings, 116° between one phenyl ring and the

pyridine ring, and 79° between the second phenyl and the pyridine ring.

The conformation of the title compound most resembles fenarimol, (3), despite the steric effects of the o-chloro substituent. The O—C_{tet}—C_{ipso}—C_{ortho} torsion angles for fenarimol are: $-41.8\,(2)$ and $139.7\,(2)°$ for the p-chlorophenyl ring, $-51.7\,(2)$ and $131.0\,(2)°$ for the o-chlorophenyl ring, and $-30.8\,(2)$ and $150.9\,(2)°$ for the pyrimidine ring. In fenarimol, the angle between the phenyl-ring planes is $84.8\,(2)°$, between the pyrimidine and o-chlorophenyl rings is $91.2\,(2)°$, and between the pyrimidine and p-chlorophenyl rings is $82.4\,(2)°$.

There is an intermolecular hydrogen bond [O(1)···N(1^i) 2.847(4) Å and O(1)—H···N(1^i) 161(5)°; symmetry code: (i) $x - 1$, y, z].
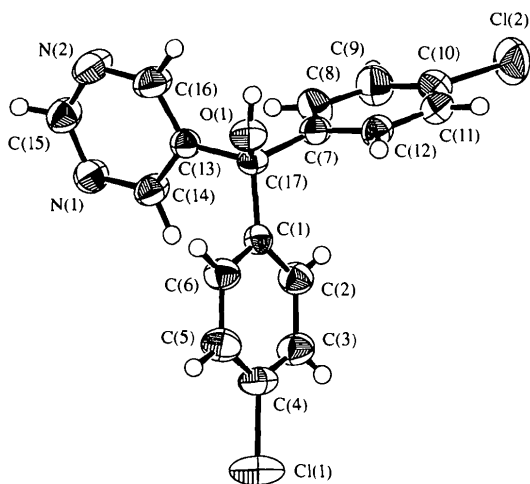

Fig. 1. The molecular structure of the title compound. Ellipsoids are shown at the 30% probability level.

Experimental

The title compound, a gift from Eli Lilly and Co (Dr M. Niedenthal), proved difficult to crystallize as good single crystals. The best crystals were obtained by evaporation of a 1:1 ethanol–chloroform solution.

Crystal data

$C_{17}H_{12}Cl_2N_2O$
$M_r = 331.20$
Monoclinic
$P2_1/n$
$a = 6.5872\,(6)$ Å
$b = 16.060\,(2)$ Å
$c = 14.876\,(1)$ Å
$\beta = 98.759\,(6)°$
$V = 1555.4\,(6)$ Å^3
$Z = 4$
$D_x = 1.409$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 25 reflections
$\theta = 36.9–40.0°$
$\mu = 3.782$ mm^{-1}
$T = 293$ K
Plate
$0.35 \times 0.25 \times 0.10$ mm
Colorless

Data collection

Rigaku AFC-5R diffractometer
θ–2θ scans
Absorption correction:
  ψ scan (TEXSAN;
  Molecular Structure Corporation, 1985)
  $T_{min} = 0.366$, $T_{max} = 0.685$
2952 measured reflections
2561 independent reflections

2000 reflections with $I > 2\sigma(I)$
$R_{int} = 0.15$
$\theta_{max} = 62°$
$h = 0 \rightarrow 7$
$k = 0 \rightarrow 18$
$l = -17 \rightarrow 16$
3 standard reflections
  every 150 reflections
  intensity decay: none

Refinement

Refinement on F
$R = 0.072$
$wR = 0.098$
$S = 1.000$
2000 reflections
202 parameters
H atoms: see below
$w = 1/(0.02726 + 0.08651F_o + 0.00329F_o^2)$

$(\Delta/\sigma)_{max} = 0.04$
$\Delta\rho_{max} = 0.429$ e Å^{-3}
$\Delta\rho_{min} = -0.451$ e Å^{-3}
Extinction correction: none
Scattering factors from International Tables for Crystallography (Vol. C)

Table 1. Selected torsion angles ($°$)

O(1)—C(17)—C(1)—C(6)	37.1 (4)
O(1)—C(17)—C(1)—C(2)	−147.3 (3)
O(1)—C(17)—C(7)—C(8)	−137.7 (3)
O(1)—C(17)—C(7)—C(12)	46.7 (4)
O(1)—C(17)—C(13)—C(14)	−137.1 (3)
O(1)—C(17)—C(13)—C(16)	39.0 (4)

An ω-scan width of $(1.15 + 0.30\tan\theta)°$, an ω-scan rate of $6°$ min^{-1} and background counts at the beginning and end of each scan, each for 50% of the total scan time, were used for data collection. Data were collected to the maximum 2θ value allowed by the instrument. The weak reflections, $I < 6\sigma(I)$, were rescanned (maximum of three rescans) and the counts accumulated to ensure good counting statistics. The intensity data were corrected for Lorentz and polarization effects, and for absorption. The structure was solved using PHASE (Calabrese, 1972) and DIRDIF (Beurskens, 1984) in the TEXSAN package (Molecular Structure Corporation, 1985) and refined by full-matrix least-squares methods. All the non-H atoms were refined anisotropically. The H atoms were introduced at calculated positions (C—H 0.96 Å) except for the hydroxyl H atom which was located from a difference Fourier map. H atoms were refined as riding on the corresponding C atoms.

Data collection: MSC/AFC Diffractometer Control Software (Molecular Structure Corporation, 1988). Cell refinement: MSC/AFC Diffractometer Control Software. Data reduction: TEXSAN (Molecular Structure Corporation, 1985). Program(s) used to refine structure: CAOS (Camalli & Spagna, 1994). Molecular graphics: VIEW (Carrell, 1976). Software used to prepare material for publication: CAOS.

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Comment

The vital roles played by glycoprotein glycans in many biological processes are well documented (Lis & Sharon, 1993). As part of our systematic study aimed at determining the three-dimensional structure of the linkage region in *N*-glycoproteins, we have previously described the crystal structure of the simplest model compound, *viz.* β-1-*N*-acetamido-D-glucopyranose (Sriram *et al.*, 1997). In continuation of this study, we report here on the crystal structure of the title compound, (I), chosen as an interesting hydrophobic analogue.

(I)
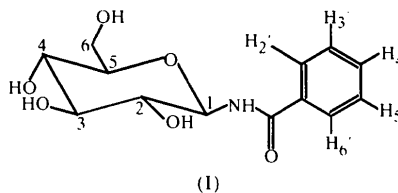
The *ORTEP*II (Johnson, 1976) plot of the benzamido derivative, giving the numbering scheme, is shown in Fig. 1. The pyranose ring adopts a $^4C_1(D)$ chair conformation, with the values of the puckering parameters (Cremer & Pople, 1975) being $Q = 0.566\,(4)$ Å, $\theta = 173.4\,(4)°$ and $\varphi = 174\,(4)°$. The amide proton is *anti* with respect to its anomeric proton, with the H1—C1—N1—H1N torsion angle being $-151°$. The benzamido group exists in the *Z-anti* conformation (H1N—N1—C1′—O1′ = 171°). The above features, together with the pyranose ring bond lengths and angles and dihedral angles, are similar to those observed for the acetamido compound (Sriram *et al.*, 1997).

β-1-*N*-Benzamido-D-glucopyranose

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Fig. 1. *ORTEP*II (Johnson, 1976) plot, showing the molecular structure and atom-numbering scheme of (I). Displacement ellipsoids are drawn at the 50% probability level. H atoms are omitted for clarity.

Abstract

In the title compound, 1-benzamido-β-D-glucopyranose, $C_{13}H_{17}NO_6$, the pyranose ring adopts the $^4C_1(D)$ conformation and the *N*-acetyl group exists in the *Z-anti* conformation. The primary alcohol group is disordered between the two permitted orientations, *gg* and *gt*.
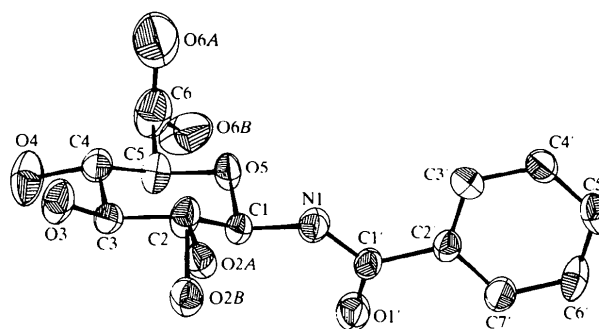
The most interesting variation noticed in the crystal structure is the twofold disorder observed for the oxygen at C6. The partial occupancies for O6*A* and O6*B* are 0.71 (1) and 0.29 (1), respectively. The primary